

US EPA ARCHIVE DOCUMENT

**WASTE CHARACTERIZATION REPORT
U.S. ANTIMONY CORPORATION
THOMPSON FALLS, MONTANA
EPA ID: MTD050261833**

Final Report - Revised

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Prepared for:

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1. Record Sampling Trip Report
U.S. Antimony Corporation Thompson Falls, Montana

1.A Overview

Production Process: Antimony Oxide

Date of Sampling Event: September 23, 1999

Dynamac Personnel: Josh Hannah, Craig Markowitz, Darline Terrell-Tyson, Frank Willford

EPA Personnel: Bob Maxey

U.S. Antimony Corporation Personnel: Mike Floersch, John Lawrence

Schedule and Procedures

8:45 a.m. EPA and Dynamac personnel proceeded to the conference room to discuss sampling issues and the RCRA program with U.S. Antimony Corporation personnel.

8:50 a.m. Mr. Bob Maxey (EPA) explained the RCRA program and presented U.S. Antimony Corporation with copies of 40 CFR parts 260 to 265 and parts 266 to 299. Mr. Maxey gave a brief overview of the listing determination process and answered RCRA regulatory questions for the facility personnel.

Mr. Mike Floersch and Mr. John Lawrence explained that U.S. Antimony Corporation has been working with the Montana DEQ and is constructing a lined containment area to store a pile of Bevill exempt mining slag that contains leachable lead. They also mentioned that the facility has been doing quarterly groundwater well monitoring for the past twenty years. Their analyses indicate that the upstream baseline contain antimony levels of over 100 times the drinking water standards.

Mr. Bob Maxey described the purpose of the Record Sampling and the listing determination process to the U.S. Antimony Corporation personnel. The wastes to be sampled were discussed.

10:25 a.m. Sampling equipment was cleaned and prepared for sample collection. Equipment blank samples were collected.

- 11:00 a.m. Began collecting the following samples: Furnace Refractory (RIN 4), Oxidation Furnace Baghouse Filters (RIN 5), Reduction Furnace Baghouse Filters (RIN 5), Reduction Furnace Slag (Sb <5%) (RIN 3), Reduction Furnace Slag (5% < Sb < 10%) (RIN 3).
- 12:45 p.m. Finished sampling. Packaged samples in ice coolers: taped over sample labels, wrapped glass jars in bubble wrap, double bagged samples in zip-lock bags. Inserted temperature blanks into coolers.
- 1:45 p.m. Split samples were relinquished to Mr. Mike Floersch.
- 1:50 p.m. Dynamac personnel departed the site and drove to Spokane, WA.
- 3:00 p.m. Arrived in Spokane, WA, bought ice at a gas station, iced down the sample coolers and taped all coolers for shipment.
- 3:20 p.m. Sample coolers dropped off at FedEx for overnight shipment to laboratory.

1.B Residuals Sampled

The following list of residuals were sampled while at the U.S. Antimony Corporation facility (RINs correspond to the RCRA §3007 questionnaire and follow-up responses):

- Furnace Refractory (RIN 4)
- Oxidation Furnace Baghouse Filters (RIN 5)
- Reduction Furnace Baghouse Filters (RIN 5)
- Reduction Furnace Slag (Sb < 5%) (RIN 3)
- Reduction Furnace Slag (5% < Sb < 10%) (RIN 3)

The following is a list of QA/QC samples collected at the U.S. Antimony Corporation facility:

- MS/MSD
- Field Duplicate
- Equipment Blank
- Temperature Blank

1.C Sample Collection

The weather during the sampling was sunny and warm. All of the sample materials were contained in sealed drums.

U.S. Antimony Corporation used the sample jars, labels, and Chain-of-Custody forms brought by Dynamac for their split samples. All sample containers requiring preservative were pre-preserved by the laboratory and sent to the sampling location.

All personnel participating in the sampling event were required to wear hard hats, and safety shoes. Rubber gloves were worn by personnel handling the samples and/or sample containers.

No photographs were taken at the U.S. Antimony Corporation facility.

1.C.1 Equipment Blank, Sample ID # AC-1-AO-04

A QA/QC equipment blank sample was collected in U.S. Antimony Corporation's laboratory. Prior to collecting the equipment blank sample the stainless steel sampling bucket was decontaminated in the following manner: The stainless steel bucket was given a gross decontamination scrub with distilled water andalconox soap, rinsed with distilled water, rinsed with a dilute nitric acid solution and then given a final rinse with deionized water.

Mr. Josh Hannah began collecting the equipment blank sample at 10:45 a.m. The decontaminated stainless steel sampling bucket was filled with deionized water. The deionized water was then poured into sample containers. The split samples for U.S. Antimony was also collected at this time. See Table 1 (Appendix D) and the footnote for a description of the sample volumes collected. EPA, Dynamac and U.S. Antimony Corporation personnel observed. Once filled, the sample containers were rinsed with water, dried with a paper towel and labeled. They were then taken to the sample staging area where tape was placed over the labels and the containers were sealed with custody seals and placed in coolers.

1.C.2 Furnace Refractory (RIN 4), Sample ID # AC-1-AO-02

This intermittent waste stream is generated when a furnace requires rebricking, approximately every 6 to 18 months. The furnace refractory is made up of used kiln brick from the furnaces. This residual varies in size from sand sized particles to boulder sized particles.

Mr. Josh Hannah began collecting the sample at 11:45 p.m. EPA, Dynamac and U.S. Antimony Corporation personnel observed the sampling. One 55 gallon drum of slag contained ground up, fairly uniform size pieces of slag. Slag from this drum was placed into a bucket using a trowel and mixed together. The samples were then taken from the bucket and placed into eight ounce jar sample containers. Dynamac's sample jars and the split sample jars were filled. See Table 1 (Appendix D) for a description of the sample volumes collected. EPA, Dynamac and U.S. Antimony Corporation personnel observed. The remaining slag in the bucket was returned to the drum and the drum was resealed. Once filled, the sample containers were labeled and taken to the sample staging area where tape was placed over the labels. The containers were then sealed with custody seals and placed in coolers.

The Furnace Refractory sample was a gray solid consisting of small pieces and sandlike material.

1.C.3 Oxidation Furnace Baghouse Filters (RIN 5), Sample ID # AC-1-AO-03

The baghouse filters are wet-cut from their cages in a wetting tank. The liquid from the wetting tank is sent to the reduction furnace. The filters are sent to a sister plant in Mexico where they are burned.

Mr. Josh Hannah began collecting the sample at 11:00 p.m. The baghouse filters were sampled from drums. The samples consisted of cutting off a large section of the filter and placing it in a zip-loc bag with the residual material that was still attached to the filter. EPA, Dynamac and U.S. Antimony Corporation personnel observed the sampling.

Dynamac's sample containers were filled first and then U.S. Antimony Corporation's split sample containers were filled. See Table 1 (Appendix D) for a description of the sample volumes collected. Once filled, the sample containers were labeled. They were then taken to the sample staging area where tape was placed over the labels and the containers were sealed with custody seals and placed in coolers.

The Oxidation Furnace Baghouse Filter sample consisted of a piece of baghouse filter coated with a white dust. These bags were moist.

1.C.4 Reduction Furnace Baghouse Filters (RIN 5), Sample ID # AC-1-AO-07

The baghouse filters are wet-cut from their cages in a wetting tank. The liquid from the wetting tank is sent to the reduction furnace. The filters are sent to a sister plant in Mexico where they are burned.

Mr. Josh Hannah began collecting the sample at 11:20 p.m. The baghouse filters were sampled from drums. The samples consisted of cutting off a large section of the filter and placing it in a zip-loc bag with the residual material that was still attached to the filter. EPA, Dynamac and U.S. Antimony Corporation personnel observed the sampling.

Dynamac's sample containers were filled first and then U.S. Antimony Corporation's split sample containers were filled. See Table 1 (Appendix D) for a description of the sample volumes collected. Once filled, the sample containers were labeled. They were then taken to the sample staging area where tape was placed over the labels and the containers were sealed with custody seals and placed in coolers.

The Reduction Furnace Baghouse Filter sample consisted of a piece of baghouse filter coated with a gray/black dust. These bags contained more dust than the oxidation furnace baghouse filters. These bags were moist.

1.C.5 Reduction Furnace Slag (Sb < 5%)(RIN 3), Sample ID # AC-1-AO-01

This intermittent waste stream is generated in the reduction furnace. The antimony levels in the slag range from less than five percent to greater than ten percent. The slag is segregated based on antimony content: greater than ten percent ($>10\%$), five to ten percent ($5\% - 10\%$), and less than five percent ($< 5\%$). If the antimony content is greater than ten percent ($>10\%$) the slag is recycled back into the furnace. If the slag is less than five percent ($< 5\%$) antimony or between five and ten percent ($5\% - 10\%$), it remains in containers until the antimony market makes it economically viable to recycle the slag back through the furnace. The amount of slag generated is approximately 20 metric tons per year.

A drum containing Reduction Furnace slag with antimony content of less than 5% was opened. Mr. Josh Hannah began collecting the sample at 12:15 p.m. EPA, Dynamac and U.S. Antimony Corporation personnel observed the sampling. Some of the material was placed into a stainless steel bucket using a trowel and mixed. The samples were then taken from the bucket and placed into eight ounce jars. The remaining slag in the bucket was returned to the drum and the drum was resealed. Dynamac's sample containers were filled from the material in the stainless steel bucket and then U.S. Antimony Corporation sample containers were filled. See Table 1 (Appendix D) for a description of the sample volumes collected. Once filled, the sample containers were labeled. They were then taken to the sample staging area where tape was placed over the labels and the containers were sealed with custody seals and placed in coolers.

The Reduction Furnace slag sample was a dark gray solid consisting of pieces approximately $3/4$ of an inch in diameter.

1.C.6 Field Duplicate, Sample ID # AC-1-AO-5

A QA/QC Field Duplicate of the Reduction Furnace slag ($Sb < 5\%$) was collected from the same material in the stainless steel bucket as described above. Mr. Josh Hannah began collecting the duplicate sample at 12:15 p.m. EPA, Dynamac and U.S. Antimony Corporation personnel observed the sampling. All sampling conditions, containers and methods were identical as the Reduction Furnace slag ($Sb < 5\%$) sample.

1.C.7 MS/MSD, Sample ID #s AC-1-AO-01-MS and AC-1-AO-01-MSD

A QA/QC Matrix Spike and Matrix Spike Duplicate were collected from the same material in the stainless steel bucket as the Reduction Furnace slag ($Sb < 5\%$) sample. Mr. Josh Hannah collected the duplicate sample immediately after collecting U.S. Antimony Corporation's Reduction Furnace slag sample. EPA, Dynamac and U.S. Antimony Corporation personnel observed the sampling. All sampling conditions, containers and methods were identical to the Reduction Furnace slag ($Sb < 5\%$) sample.

1.C.8 Reduction Furnace Slag ($5\% < Sb < 10\%$)(RIN 3), Sample ID # AC-1-AO-06

This intermittent waste stream is generated in the reduction furnace. The antimony levels in the slag range from less than five percent to greater than ten percent. The slag is segregated based on

antimony content: greater than ten percent (>10%), five to ten percent (5% - 10%), and less than five percent (< 5%). If the antimony content is greater than ten percent (>10%) the slag is recycled back into the furnace. If the slag is less than five percent (< 5%) antimony or between five and ten percent (5% - 10%), it remains in containers until the antimony market makes it economically viable to recycle the slag back through the furnace. The amount of slag generated is approximately 20 metric tons per year.

A drum containing Reduction Furnace slag with antimony content between 5% and 10% was opened. Mr. Josh Hannah began collecting the sample at 12:30 p.m. EPA, Dynamac and U.S. Antimony Corporation personnel observed the sampling. Some of the material was placed into a bucket using a trowel and mixed together. The samples were then taken from the bucket and placed into eight ounce jars. The remaining slag in the bucket was returned to the drum and the drum was resealed.

Dynamac's sample containers were filled from the material in the stainless steel bucket and then U.S. Antimony Corporation sample containers were filled. See Table 1 (Appendix D) for a description of the sample volumes collected. Once filled, the sample containers were labeled. They were then taken to the sample staging area where tape was placed over the labels and the containers were sealed with custody seals and placed in coolers.

The Reduction Furnace slag sample was a dark gray solid consisting of pieces approximately 3/4 of an inch in diameter.

1.D Deviations from Sampling and Analysis Plan

There were some deviations from the Sampling and Analysis Plan. The first to note is that an extra sample was collected. It was determined that an extra baghouse filter sample should be collected after it became apparent that there were two different baghouse filter residuals. One baghouse filter cleans the air associated with the oxidation furnace (AC-1-AO-03) the other filters the air associated with the reduction furnace (AC-1-AO-07).

The second deviation to note is that instead of collecting two samples of the Refractory Furnace Slag based on particle size (small size particle and large particle size), it was determined to collect two slag samples based on antimony content (< 5% and 5% - 10%). U.S. Antimony handles their Refractory Furnace Slag slightly different than was anticipated. Instead of having large and small size slag particles, the batches of the slag is first ground up into small, fairly similar size pieces prior to being sealed in 55 gallon drums.

1.E Packaging and Shipping

One temperature blank sample vial was placed inside each of the sample coolers to be shipped back to the laboratory.

After all of the sampling had been completed, the samples were taken to the parking lot outside the

U.S. Antimony Corporation office building where they were repackaged. The final packaging included wrapping all glass containers in bubble wrap and double sealing all sample containers in zip-lock bags. Double layered garbage bags were placed inside ice coolers. The sealed sample containers were then placed inside the garbage bags. A Chain of Custody form was filled out for each ice cooler, sealed in a zip-lock bag and taped to the inside lid of the cooler.

Dynamac personnel then departed the U.S. Antimony Corporation facility and went to Spokane, WA. They arrived in Spokane, WA at 3:00 p.m. and stopped at a gas station to purchase bags of ice. Ice was placed inside the garbage bags covering the sample containers sealed in zip-lock bags to keep the samples cool. The coolers were then securely taped for shipment, transported to a local Federal Express location and shipped via overnight delivery to APPL, Inc. in Fresno, California for analysis.

2. DRAFT DATA VALIDATION REPORT UNITED STATES ANTIMONY CORPORATION RECORD SAMPLING

2.A Overview

Seven samples were collected at United States Antimony Corporation during a site visit on September 23, 1999: three furnace slag samples, one furnace refractory sample, two filter samples, and an equipment blank. The sample set included one field duplicate pair. The samples were analyzed for the analytes specified in the United States Antimony Corporation Site Sampling and Analysis Plan for Record Sampling Under the Inorganic Listing Determination, dated September 22, 1999. The methods used for these analyses are presented in Table 1. The laboratory did not deviate from the methods as written except for the following: (i) for hexavalent chromium analysis of solid samples, the laboratory extracted the samples 1:1 with deionized water as the samples were too alkaline for the requested alkaline digestion (method 3060); and (ii) for TCLP hexavalent chromium, the laboratory used deionized water as the extraction fluid. Note that unlike for previous sites under the Inorganic Listing Determination, the reported SPLP hexavalent chromium results for these samples were generated using extraction fluid #1 (and not deionized water).

The data were reviewed according to the procedures outlined in the Quality Assurance Project Plan for Characterization Sampling for Inorganic Chemicals Listing Determination. The text of this report addresses only those problems affecting usability.

The analytical results for the U.S. Antimony samples, with the applied data qualifiers, are presented in Table 2. The results for the method blanks and leachate blanks prepared at the laboratory are also presented.

2.B Data Validation Summary

The samples identified in the SAP were collected in the volumes specified in the SAP.

2.B.1 Holding Times: All extractions and analyses were completed within the required holding times with the exception of hexavalent chromium in AC-1-AO-04. The result was qualified as estimated with a low bias (UL for non-detect result) in this sample.

The dates of analysis for each sample are detailed in the attached worksheet.

2.B.2 Instrument Calibration: All QC requirements for instrument calibration were met for all analyses. Calibration curves for CVAA, AA, and colorimetric analyses had correlation coefficients ≥ 0.995 . All initial calibration verification samples were within the QC limits of 90-110%, and all continuing calibration verification samples were within the QC limits of 90-110% for ICP and 80-120% for CVAA, AA, and colorimetric analytes.

2.B.3 Blank Analysis Results: No target analytes were detected in the method and leachate blanks. Antimony and calcium were detectable in the equipment blank, AC-1-AO-04 (see Table 2). No qualification of data was necessary as the levels of these analytes in the samples either greatly exceeded the levels in the blank or were non-detectable.

2.B.4 Interference Check Sample (ICP Analyses): The recovery of spiked analytes in the Interference Check Sample (ICS) was within the QC limits of 80-120% for all analytes.

2.B.5 Matrix Spike/Matrix Spike Duplicate Results: The results of matrix spike/matrix spike duplicate analyses are presented in Tables 3 (total inorganics), 4 (TCLP), and 5 (SPLP). The recovery of analytes in the matrix spike and matrix spike duplicate samples was within the QC limits of 75-125%, and the duplication was within the QC limits of 0-25% relative percent difference, with the following exceptions: total antimony, arsenic, barium, boron, copper, iron, lead, manganese, silver, sodium, titanium, and zinc; TCLP boron; and SPLP antimony, arsenic, beryllium, boron, cadmium, iron, nickel, sodium, thallium, titanium, and vanadium. The data qualifiers that were applied to data as a result of these QC exceedances are detailed in the footnotes to the respective tables. Data qualifiers for these QC exceedances were only applied to samples of similar matrix to the sample used for matrix spike analyses (i.e., qualifiers were only applied to samples AC-1-AO-01, AC-1-AO-05, and AC-1-AO-06). For total arsenic, barium, boron, iron, sodium, titanium, and zinc, and SPLP antimony, arsenic, boron, sodium, and vanadium, no action was necessary as the sample concentration greatly exceeded the spike concentration. The data qualifier used to qualify total iron results was determined based on the results of duplicate analyses of sample AC-1-AO-01 which demonstrated poor relative percent duplication.

For some analytes, the laboratory did not conduct matrix spike analyses but did conduct duplicate analyses; these results are presented in Table 3.

2.B.6 Post-Digestion Spike Results: Although the laboratory conducted post-digestion spike analyses for all ICP analytes, post-digestion spike results were only reported for analytes for which matrix spike and matrix spike duplicate recovery failed (and the sample result was less than four times the spike amount). For this sample set, post-digestion spike recovery was reported for SPLP beryllium, cadmium, iron, nickel, thallium, and titanium. For SPLP beryllium, cadmium, iron, and nickel, the recovery was below the QC limits of 75-125% (see attached worksheet). No additional qualification of data was necessary as all affected sample results had previously been qualified due to poor matrix spike recovery.

2.B.7 Laboratory Control Sample Results: The recovery of analytes in the laboratory control samples is presented in Tables 6 (aqueous sample), 7 (solid sample), 8 (TCLP sample), and 9 (SPLP sample). The recovery was within the QC limits of 80-120% for all analytes except TCLP zinc (extraction fluid #2), and SPLP barium and boron. The actions that were taken as a result of this QC exceedance are detailed in the respective tables.

2.B.8 ICP Serial Dilution Results (ICP Analyses): The laboratory conducted serial dilution

analyses for ICP analytes only in cases for which matrix spike recovery failed, the sample result was greater than 4 times the spike added, and the sample result was greater than 50 times the reporting limit. For this sample, serial dilution was required for total antimony, arsenic, barium, iron, titanium, and zinc, and SPLP antimony, arsenic, boron, and vanadium. However, the samples were run at dilutions of 1:10, 1:100, and 1:1000 to get sample results within calibration range. Therefore, the results of ICP serial dilution cannot be evaluated.

2.B.9 Target Detection Limits: The target detection limits identified in the QAPP were met for all analytes except as detailed in the table below:

Target Compound	Target Detection Limit (mg/L)	Achieved Method Detection Limit (mg/L) ¹
Wastewaters		
Arsenic	0.002	0.00237 (0.005)
Thallium	0.0005	0.00216 (0.005)

¹ Quantitation limit used for reporting in parentheses.

Also, because of the high levels of antimony and sodium in the solid samples, several samples had to be diluted before analysis for total metals, resulting in elevated detection limits for total metal analytes.

2.B.10 Field Duplicate Results: The sampling trip included one field duplicate pair: AC-1-AO-01 and AC-1-AO-05. The relative percent difference in the results for all detected analytes is presented in Table 10. All results were within the QC limits of 0-50 percent relative difference with the exception of total antimony, TCLP nickel and titanium, and SPLP barium and titanium. The actions that were taken as a result of these QC exceedances are detailed in Table 10.

Attachments:

1. Glossary of Data Qualifier Codes (Appendix A)
2. List of Acronyms (Appendix B)
3. Data Validation Worksheet (Appendix C)

Table 1. Digestion and Analysis Methods Used for U.S. Antimony Samples. ¹

Target Analyte	Digestion Method ²	Analysis Method
Antimony	3010/3050	6010 (ICP)
Arsenic	3010/3050	6010 (ICP)
Barium	3010/3050	6010 (ICP)
Beryllium	3010/3050	6010 (ICP)
Boron	3010/3050	6010 (ICP)
Cadmium	3010/3050	6010 (ICP)
Calcium	3010/3050	6010 (ICP)
Chromium	3010/3050	6010 (ICP)
Chromium ⁶⁺	7196/3060	7196 (colorimetric)
Cobalt	3010/3050	6010 (ICP)
Copper	3010/3050	6010 (ICP)
Iron	3010/3050	6010 (ICP)
Lead	3010/3050	6010 (ICP)
Manganese	3010/3050	6010 (ICP)
Mercury	7470/7471	7470/7471 ³ (CVAA)
Nickel	3010/3050	6010 (ICP)
Potassium	3010/3050	7610 (Flame AA)
Selenium	3010/3050	6010 (ICP)
Silver	3010/3050	6010 (ICP)
Sodium	3010/3050	7770 (Flame AA)
Thallium	3010/3050	6010 (ICP)
Titanium	3010/3050	6010 (ICP)
Vanadium	3010/3050	6010 (ICP)
Zinc	3010/3050	6010 (ICP)
TCLP	1311 ⁴	N/A
SPLP	1312 ⁴	N/A
pH	N/A	EPA 150.1/ SW-846 9045 ³
Oxidation/reduction (mV)	N/A	ASTM D1498-93
Specific gravity	N/A	ASTM D854-83
Moisture content	N/A	CLP 3/90

¹ All methods are SW-846 methods unless otherwise indicated.

² The first method listed is the digestion method for aqueous matrices (leachates); the second method listed is for solid matrices.

³ The first method is for aqueous matrices (leachates); the second method is for solid matrices.

⁴ Following the leaching procedure, the leachates were digested according to the digestion method listed for each analyte.

Table 2. United States Antimony Corporation - Sample Results ¹

Laboratory Sample Number	AP84021						AP84022					
EPA Sample Number	AC-1-AO-01						AC-1-AO-05					
Date Sampled	09/23/1999						09/23/1999					
Sample Description	Reduction furnace slag (Sb < 5%)						Reduction furnace slag (Sb < 5%) - field duplicate					
Result Type	Total		TCLP		SPLP		Total		TCLP		SPLP	
Units	mg/kg		mg/L		mg/L		mg/kg		mg/L		mg/L	
Target Analyte	Result	DQ ²	Result	DQ	Result	DQ	Result	DQ	Result	DQ	Result	DQ
Inorganics												
Antimony	11500	J	55.8		114		19600	J	88.4		113	
Arsenic	301		2.0		2.93		349		2.3		2.75	
Barium	294		<2	U	<2	U	311		<2	U	0.135	K
Beryllium	<20	U	<0.02	U	0.0034	L	<20	U	<0.02	U	0.0032	L
Boron	<500	U	9.8	K	9.27		<500	U	15.1	K	9.72	J
Cadmium	<50	U	<0.05	U	<0.005	UL	<50	U	<0.05	U	<0.005	UL
Calcium	8130		20.1		3.42		8730		19.3		3.63	
Chromium	<50	U	<0.05	U	<0.005	U	<50	U	<0.05	U	<0.005	U
Chromium ⁶⁺	<0.02 ³	U	<0.50	U	<0.50	U	<0.02 ³	U	<0.02	U	<0.50	U
Cobalt	<50	U	<0.05	U	0.0061		<50	U	<0.05	U	0.0072	
Copper	52.2	K	<0.25	U	0.0087		65.4	K	<0.25	U	0.0080	
Iron	13600		1.3		0.662	L	14800		1.4		0.866	L
Lead	135	J	<0.5	U	<0.005	U	219	J	<0.5	U	<0.005	U
Manganese	160	K	<0.05	U	<0.005	U	174	K	<0.05	U	<0.005	U
Mercury	<0.1	U	<0.002	U	<0.0002	U	<0.1	U	<0.002	U	<0.0002	U
Nickel	<50	U	<0.2	U	<0.005	UL	<50	U	<0.2	U	<0.005	UL
Potassium	2020		89.6		87.6		2310		90.5		85.9	
Selenium	<50	U	0.6		0.550		<50	U	0.8		0.493	
Silver	<10	U	<0.1	U	<0.001	U	<10	U	<0.1	U	<0.001	U
Sodium	321000		N/A		13000		329000		N/A		13800	
Thallium	<200	U	<2	U	<0.005	UL	<200	U	<2	U	<0.005	UL
Titanium	2440		0.067		<0.005	UL	2650		<0.05	U	0.0063	L
Vanadium	<50	U	1.3		1.14		<50	U	1.4		1.2	
Zinc	<500	U	<3	U	<0.05	U	<500	U	<3	U	<0.100	U
Physical Properties												
Specific gravity (unitless)	2.7		N/A		N/A		2.8		N/A		N/A	
Moisture content, %	<2	U	N/A		N/A		<2	U	N/A		N/A	

Table 2. United States Antimony Corporation - Sample Results ¹ (continued).

Laboratory Sample Number	AP84021						AP84022					
EPA Sample Number	AC-1-AO-01						AC-1-AO-05					
Date Sampled	09/23/1999						09/23/1999					
Sample Description	Reduction furnace slag (Sb < 5%)						Reduction furnace slag (Sb < 5%) - field duplicate					
Result Type	Total		TCLP		SPLP		Total		TCLP		SPLP	
Units	mg/kg		mg/L		mg/L		mg/kg		mg/L		mg/L	
Target Analyte	Result	DQ ²	Result	DQ	Result	DQ	Result	DQ	Result	DQ	Result	DQ
Final pH of leachate	N/A		11.6		12.9		N/A		11.7		12.7	

Table 2. United States Antimony Corporation - Sample Results ¹ (continued).

Laboratory Sample Number	AP84020						AP84017					
EPA Sample Number	AC-1-AO-02						AC-1-AO-03					
Date Sampled	09/23/1999						09/23/1999					
Sample Description	Furnace refractory						Oxidation furnace baghouse filters					
Result Type	Total		TCLP		SPLP		Total		TCLP		SPLP	
Units	mg/kg		mg/L		mg/L		mg/kg		mg/L		mg/L	
Target Analyte	Result	DQ ²	Result	DQ	Result	DQ	Result	DQ	Result	DQ	Result	DQ
Inorganics												
Antimony	152000		10.9		62.3		150000		9.9		4.31	
Arsenic	<250	U	<0.5	U	0.285		<250	U	<0.5	U	0.0899	
Barium	<250	U	<2	U	<2	U	<250	U	<2	U	<2	U
Beryllium	<100	U	<0.02	U	<0.002	U	<100	U	<0.02	U	<0.002	U
Boron	<2500	U	4.3		3.760		<2500	U	<2	U	0.198	
Cadmium	<250	U	<0.05	U	<0.005	U	<250	U	0.3		0.266	
Calcium	<5000	U	13.3		1.97		5650		<2	U	0.404	
Chromium	<250	U	<0.05	U	<0.005	U	<250	U	<0.05	U	<0.005	U
Chromium ⁶⁺	<0.02 ³	U	<0.02	U	<0.02	U	<0.02 ³	U	<0.02	U	<0.02	U
Cobalt	653		0.1		0.0302		<250	U	<0.05	U	<0.005	U
Copper	3080		<0.25	U	0.0836		<250	U	<0.25	U	0.0177	
Iron	14700		69.8		0.423		<2500	U	<1	U	0.0807	
Lead	9250		47.4		0.861		<250	U	2.8		0.984	
Manganese	<250	U	1.2		0.0155		<250	U	<0.05	U	0.0246	
Mercury	0.3		<0.002	U	<0.0002	U	0.1		<0.002	U	<0.0002	U
Nickel	1310		0.2		0.0665		<250	U	<0.2	U	0.0050	
Potassium	289		<10	U	1.5		126		<10	U	<1	U
Selenium	<250	U	<0.5	U	0.0060		<250	U	<0.5	U	0.0193	
Silver	851		<0.1	U	0.0142		<50	U	<0.1	U	<0.001	U
Sodium	1170		N/A		80.1		294		N/A		13.3	
Thallium	<1000	U	<2	U	<0.005	U	<1000	U	<2	U	0.0584	
Titanium	<250	U	<0.05	U	0.0433		<250	U	<0.05	U	<0.005	U
Vanadium	<250	U	<0.05	U	<0.005	U	<250	U	<0.05	U	<0.005	U
Zinc	<2500	U	<2	U	0.143		<2500	U	<2	U	0.155	
Physical Properties												
Specific gravity (unitless)	3.6		N/A		N/A		1.3		N/A		N/A	
Moisture content, %	<2	U	N/A		N/A		24.1		N/A		N/A	

Table 2. United States Antimony Corporation - Sample Results ¹ (continued).

Laboratory Sample Number	AP84020						AP84017					
EPA Sample Number	AC-1-AO-02						AC-1-AO-03					
Date Sampled	09/23/1999						09/23/1999					
Sample Description	Furnace refractory						Oxidation furnace baghouse filters					
Result Type	Total		TCLP		SPLP		Total		TCLP		SPLP	
Units	mg/kg		mg/L		mg/L		mg/kg		mg/L		mg/L	
Target Analyte	Result	DQ ²	Result	DQ	Result	DQ	Result	DQ	Result	DQ	Result	DQ
Final pH of leachate	N/A		5.11		7.77		N/A		5.04		4.55	

Table 2. United States Antimony Corporation - Sample Results ¹ (continued).

Laboratory Sample Number	AP84023						AP84018					
EPA Sample Number	AC-1-AO-06						AC-1-AO-07					
Date Sampled	09/23/1999						09/23/1999					
Sample Description	Reduction furnace slag (5% < Sb <10%)						Reduction furnace baghouse filters					
Result Type	Total		TCLP		SPLP		Total		TCLP		SPLP	
Units	mg/kg		mg/L		mg/L		mg/kg		mg/L		mg/L	
Target Analyte	Result	DQ	Result	DQ	Result	DQ	Result	DQ	Result	DQ	Result	DQ
Inorganics												
Antimony	127000	J	110		211		145000		68.7		287	
Arsenic	478		3.1		3.81		<250	U	6.9		6.87	
Barium	257		<2	U	<0.1	U	<250	U	<2	U	<2	U
Beryllium	<100	U	<0.02	U	0.0024	L	<100	U	<0.02	U	<0.02	U
Boron	<2500	U	8.5	K	8.06	J	<2500	U	<2	U	0.662	
Cadmium	<250	U	<0.05	U	<0.005	UL	411		<0.05	U	0.869	
Calcium	9000		17.0		4.46		6880		<2	U	2.210	
Chromium	<250	U	<0.05	U	<0.005	U	<250	U	<0.05	U	<0.05	U
Chromium ⁶⁺	<0.02 ³	U	<0.20	U	<0.50	U	<0.02 ³	U	<0.02	U	<0.02	U
Cobalt	<250	U	<0.05	U	<0.005	U	<250	U	<0.05	U	<0.05	U
Copper	<250	U	<0.25	U	0.0079		270		<0.25	U	0.194	
Iron	13500		8.8		2.87	L	<2500	U	<1	U	<0.5	U
Lead	491	J	<0.5	U	<0.005	U	<250	U	<0.5	U	<0.05	U
Manganese	<250	U	<0.05	U	<0.005	U	<250	U	0.060		<0.05	U
Mercury	<0.1	U	<0.002	U	0.0003		95.2		0.026		0.370	
Nickel	<250	U	<0.2	U	<0.005	UL	<250	U	<0.2	U	<0.05	U
Potassium	1980		83.6		83.4		216		<10	U	5.1	
Selenium	<250	U	0.6		0.331		<250	U	<0.5	U	0.0614	
Silver	<100	U	<0.1	U	<0.001	U	61.0		<0.1	U	0.077	
Sodium	262000		N/A		11100		7110		N/A		244	
Thallium	<1000	U	<2	U	<0.005	UL	<1000	U	<2	U	0.0965	
Titanium	761		0.2		<0.005	UL	<250	U	<0.05	U	<0.05	U
Vanadium	<250	U	0.6		1.0		<250	U	<0.05	U	<0.05	U
Zinc	<2500	U	<3	U	<0.100	U	3440		<2	U	0.624	
Physical Properties												
Specific gravity (unitless)	2.6		N/A		N/A		0.95		N/A		N/A	
Moisture content, %	<2		N/A		N/A		5.5		N/A		N/A	

Table 2. United States Antimony Corporation - Sample Results ¹ (continued).

Laboratory Sample Number	AP84023						AP84018					
EPA Sample Number	AC-1-AO-06						AC-1-AO-07					
Date Sampled	09/23/1999						09/23/1999					
Sample Description	Reduction furnace slag (5% < Sb <10%)						Reduction furnace baghouse filters					
Result Type	Total		TCLP		SPLP		Total		TCLP		SPLP	
Units	mg/kg		mg/L		mg/L		mg/kg		mg/L		mg/L	
Target Analyte	Result	DQ	Result	DQ	Result	DQ	Result	DQ	Result	DQ	Result	DQ
Final pH of leachate	N/A		10.8		7.62		N/A		5.22		9.99	

Table 2. United States Antimony Corporation - Sample Results ¹ (continued).

Laboratory Sample Number	AP84019									
EPA Sample Number	AC-1-AO-04									
Date Sampled	09/23/1999									
Sample Description	Equipment blank		Method blank - water		Method blank - solid		Leachate blank		Leachate blank	
Result Type	Total		Total		Total		TCLP		SPLP	
Units	mg/L		mg/L		mg/kg		mg/L		mg/L	
Target Analyte	Result	DQ	Result	DQ	Result	DQ	Result	DQ	Result	DQ
Inorganics										
Antimony	0.0371		<0.005	U	<0.5	U	<0.5	U	<0.005	U
Arsenic	<0.005	U	<0.005	U	<0.5	U	<0.5	U	<0.005	U
Barium	<0.005	U	<0.005	U	<0.5	U	<2	U	<0.050	U
Beryllium	<0.002	U	<0.002	U	<0.2	U	<0.02	U	<0.002	U
Boron	<0.050	U	<0.050	U	<10	U	<2	U	<0.050	U
Cadmium	<0.005	U	<0.005	U	<0.5	U	<0.05	U	<0.005	U
Calcium	0.235		<0.100	U	<100	U	<2	U	<0.100	U
Chromium	<0.005	U	<0.005	U	<0.5	U	<0.05	U	<0.005	U
Chromium ⁶⁺	<0.02	UL	<0.020	U	<0.02	U	<0.02	U	<0.02	U
Cobalt	<0.005	U	<0.005	U	<0.5	U	<0.05	U	<0.005	U
Copper	<0.005	U	<0.005	U	<0.5	U	<0.25	U	<0.005	U
Iron	<0.05	U	<0.050	U	<5	U	<1	U	<0.050	U
Lead	<0.003	U	<0.003	U	<0.3	U	<0.5	U	<0.003	U
Manganese	<0.005	U	<0.005	U	<0.5	U	<0.05	U	<0.005	U
Mercury	<0.0002	U	<0.0002	U	<0.1	U	<0.002	U	<0.0002	U
Nickel	<0.005	U	<0.005	U	<0.5	U	<0.2	U	<0.005	U
Potassium	<1	U	<1	U	<100	U	<10	U	<1	U
Selenium	<0.005	U	<0.005	U	<0.5	U	<0.5	U	<0.005	U
Silver	<0.001	U	<0.001	U	<0.1	U	<0.2	U	<0.001	U
Sodium	<1	U	<1	U	<100	U	N/A		<1	U
Thallium	<0.005	U	<0.005	U	<2	U	<2	U	<0.005	U
Titanium	<0.005	U	<0.005	U	<0.5	U	<0.1	U	<0.005	U
Vanadium	<0.005	U	<0.005	U	<0.5	U	<0.05	U	<0.005	U
Zinc	<0.050	U	<0.050	U	<5	U	<3	U	<0.050	U
Physical Properties										
pH, pH units	3.6		N/A		N/A		N/A		N/A	
Oxidation/reduction, O/R	261		N/A		N/A		N/A		N/A	
Specific gravity (unitless)	1.0		201.9		N/A		N/A		N/A	

¹ < = Less than the reporting limit specified. N/A = Not analyzed.

² DQ = Data Qualifier.

- ³ The laboratory reported that because this sample is very alkaline, the requested alkaline digestion (SW-846 3060A) for hexavalent chromium could not be conducted. Instead the laboratory conducted a 1:1 extraction with deionized water. The result is reported as mg/L.

Table 3. Matrix Spike/Matrix Spike Duplicate Results - Total Inorganics

Analyte	Sample ID: AC-1-AO-01			Action ¹
	MS Recovery, %	MSD Recovery, %	% RPD	
Antimony	1980.6	17153.8	46.7	J - positive results ²
Arsenic	150.3	183.5	4.3	None ³
Barium	108.7	72.5	5.3	None ³
Beryllium	114.0	113.0	0.7	None
Boron	148.4	106.1	7.9	None ³
Cadmium	98.0	92.1	5.0	None
Calcium	112.9	103.5	3.5	None
Chromium	107.9	104.8	2.5	None
Chromium ⁶⁺	89.3	92.7	3.7	None
Cobalt	107.1	108.5	1.2	None
Copper	122.0	160.9	15.8	K - positive results ²
Iron	450.1	326.6	1.7	None ³
Lead	308.6	436.7	19.9	J - positive results ²
Manganese	126.9	105.6	4.9	K - positive results ²
Mercury	92.6	91.7	1.0	None
Nickel	108.9	115.5	4.5	None
Potassium	118	105	4.0	None
Selenium	93.6	77.2	13.1	None
Silver	99.7	125.9	20.2	None ⁴
Sodium	230	-68	4.6	None ³
Thallium	82.6	110.6	18.2	None
Titanium	335.6	236.3	1.9	None ³
Vanadium	105.0	100.6	2.4	None
Zinc	373.9	254.5	13.5	None ³

¹ QC limits are 75-125% recovery and 0-25% RPD (relative percent difference).

² Actions apply only to results for total analysis in samples AC-1-AO-01, AC-1-AO-05, and AC-1-AO-06.

³ No action taken as sample concentration greatly exceeded spike concentration.

⁴ No action required as total silver was not detected in the affected samples (AC-1-AO-01, AC-1-AO-05, and AC-1-AO-06).

Inorganics, Laboratory Duplicate				
Sample ID: AC-1-AO-04				
Analyte	Sample Result	Duplicate Result	% RPD	Action ¹
Specific gravity	1.00	1.00	0.0%	None

¹ QC limits are 0-25% RPD (relative percent difference).

Table 4. Matrix Spike/Matrix Spike Duplicate Results - TCLP Inorganics

Analyte	Sample ID: AC-1-AO-01			Action ¹
	MS Recovery, %	MSD Recovery, %	RPD, %	
Antimony	81.6	81.0	0.0	None
Arsenic	102.0	103.0	0.7	None
Barium	100.1	100.3	0.2	None
Beryllium	94.4	94.6	0.2	None
Boron	216.2	107.7	30.4	K - positive results ²
Cadmium	98.6	97.9	0.7	None
Calcium	101.6	102.0	0.3	None
Chromium	97.5	97.4	0.1	None
Chromium ⁶⁺	95.3	96.0	0.7	None
Cobalt	93.8	94.2	0.4	None
Copper	108.0	108.2	0.2	None
Iron	94.8	96.2	1.3	None
Lead	97.9	98.7	0.8	None
Manganese	95.2	95.3	0.1	None
Mercury	98.7	101	2.0	None
Nickel	93.4	94.0	0.6	None
Potassium	110	103	3.7	None
Selenium	100.4	101.1	0.6	None
Silver	104.1	104.9	0.8	None
Thallium	98.9	99.4	0.5	None
Titanium	95.3	95.3	0.0	None
Vanadium	101.7	101.8	0.1	None
Zinc	105.2	105.5	0.3	None

¹ QC limits are 75-125% recovery and 0-25% RPD.

² For samples AC-1-AO-01, AC-1-AO-05, and AC-1-AO-06.

Table 5. Matrix Spike/Matrix Spike Duplicate Results - SPLP Inorganics

Analyte	Sample ID: AC-1-AO-01			Action ¹
	MS Recovery, %	MSD Recovery, %	RPD, %	
Antimony	-8540.0	-8511.3	0.1	None ²
Arsenic	-56.1	-60.8	0.4	None ²
Barium	77.7	77.5	0.2	None
Beryllium	71.2	71.0	0.3	L - positive results; UL - non-detect results ³
Boron	-493.7	-497.0	0.1	None ²
Cadmium	69.9	70.9	1.3	L - positive results; UL - non-detect results ³
Calcium	77.1	76.7	0.4	None
Chromium	75.6	75.5	0.2	None
Chromium ₆₊	88.7	91.3	3.0	None
Cobalt	76.4	76.0	0.6	None
Copper	94.8	93.9	0.9	None
Iron	67.3	67.9	0.4	L - positive results; UL - non-detect results ³
Lead	77.4	77.0	0.5	None
Manganese	77.6	77.1	0.6	None
Mercury	104	107	2.9	None
Nickel	71.8	71.9	0.1	L - positive results; UL - non-detect results ³
Potassium	109	96.8	0.6	None
Selenium	110.0	107.8	0.7	None
Silver	100.9	100.9	0.1	None
Sodium	3280	2840	0.8	None ²
Thallium	73.0	73.4	0.5	L - positive results; UL - non-detect results ³
Titanium	74.0	72.3	2.3	L - positive results; UL - non-detect results ³
Vanadium	73.7	70.8	0.6	None ²
Zinc	110.3	113.1	2.5	None

¹ QC limits are 75-125% recovery and 0-25% RPD (relative percent difference).

² No action required as sample concentration greatly exceeded spike concentration.

³ Actions apply only to SPLP results in samples AC-1-AO-01, AC-1-AO-05, and

AC-1-AO-06.

Table 6. Laboratory Control Sample (LCS) Results - Inorganics Water Matrix

Analyte	LCS Recovery, % ¹		QC Limits ²	Compliant?
	LCSW-1	LCSW-2		
Antimony	97.5	99.7	80-120	Yes
Arsenic	96.8	96.8	80-120	Yes
Barium	102.3	101.9	80-120	Yes
Beryllium	99.7	99.1	80-120	Yes
Boron	103.9	94.2	80-120	Yes
Cadmium	99.6	98.7	80-120	Yes
Calcium	103.8	103.3	80-120	Yes
Chromium	100.7	99.8	80-120	Yes
Chromium ⁶⁺	98.0	99.7 (10/4/99)	80-120	Yes
Cobalt	92.9	92.4	80-120	Yes
Copper	103.0	102.7	80-120	Yes
Iron	100.5	100.7	80-120	Yes
Lead	99.0	98.0	80-120	Yes
Manganese	98.6	98.3	80-120	Yes
Mercury	98.7	100	80-120	Yes
Nickel	96.1	94.9	80-120	Yes
Potassium	100	97.9 (10/12/99)	80-120	Yes
Selenium	90.7	89.7	80-120	Yes
Silver	101.8	101.4	80-120	Yes
Sodium	93.6	93.0 (10/11/99)	80-120	Yes
Thallium	92.1	91.8	80-120	Yes
Titanium	99.9	99.6	80-120	Yes
Vanadium	103.1	102.6	80-120	Yes
Zinc	98.1	100.6	80-120	Yes

¹ Samples were analyzed 10/8/99 unless otherwise indicated in parentheses.

² QC limits reported by laboratory.

Table 7. Laboratory Control Sample (LCS) Results - Inorganics Solid Matrix

Analyte	LCS Recovery, % ¹		QC Limits ²	Compliant?
	1	2		
Antimony	100.1	98.6	80-120	Yes
Arsenic	100.0	99.7	80-120	Yes
Barium	102.7	101.3	80-120	Yes
Beryllium	101.4	100.8	80-120	Yes
Boron	102.1	115.1	80-120	Yes
Cadmium	103.4	102.0	80-120	Yes
Calcium	102.4	102.1	80-120	Yes
Chromium	103.4	102.0	80-120	Yes
Chromium ⁶⁺	98.0	100 (10/4/99)	80-120	Yes
Cobalt	99.8	100.0	80-120	Yes
Copper	100.8	104.1	80-120	Yes
Iron	104.0	103.5	80-120	Yes
Lead	102.9	101.9	80-120	Yes
Manganese	102.5	102.7	80-120	Yes
Mercury	101 (10/7/99)	99.0 (10/8/99)	80-120	Yes
Nickel	104.4	103.0	80-120	Yes
Potassium	103	109 (10/8/99)	80-120	Yes
Selenium	94.3	94.0	80-120	Yes
Silver	99.3	99.2	80-120	Yes
Sodium	93.4	97.8 (10/8/99)	80-120	Yes
Thallium	98.6	98.7	80-120	Yes
Titanium	101.5	102.3	80-120	Yes
Vanadium	103.0	103.3	80-120	Yes
Zinc	103.4	101.7	80-120	Yes

¹ Samples were analyzed 10/15/99 unless otherwise indicated in parentheses.

² QC limits reported by laboratory.

Table 8. Laboratory Control Sample (LCS) Results - TCLP Inorganics

Analyte	LCS Recovery, % (Extraction Fluid #1) ¹	QC Limits ²	Compliant?
Antimony	100.1, 101.2	80-120	Yes
Arsenic	99.7, 99.8	80-120	Yes
Barium	116.1, 116.4	80-120	Yes
Beryllium	97.0, 97.5	80-120	Yes
Boron	103.8, 104.3	80-120	Yes
Cadmium	99.0, 99.6	80-120	Yes
Calcium	103.8, 104.2	80-120	Yes
Chromium	99.9, 100.0	80-120	Yes
Chromium ⁶⁺	94.7, 96.0 ³	80-120	Yes
Cobalt	93.5, 94.0	80-120	Yes
Copper	106.6, 106.9	80-120	Yes
Iron	98.7, 98.8	80-120	Yes
Lead	98.4, 98.8	80-120	Yes
Manganese	100.1, 100.3	80-120	Yes
Mercury	102, 102 (10/7/99)	80-120	Yes
Nickel	94.1, 94.9	80-120	Yes
Potassium	108	80-120	Yes
Selenium	97.0, 97.7	80-120	Yes
Silver	103.8, 104.0	80-120	Yes
Thallium	95.6, 96.0	80-120	Yes
Titanium	104.5, 104.9	80-120	Yes
Vanadium	102.6, 102.6	80-120	Yes
Zinc	113.2, 114.0	80-120	Yes
Analyte	LCS Recovery, % (Extraction Fluid #2) ⁴	QC Limits ²	Compliant?
Antimony	99.8	80-120	Yes
Arsenic	102.2	80-120	Yes
Barium	109.1	80-120	Yes
Beryllium	96.3	80-120	Yes
Boron	105.6	80-120	Yes
Cadmium	100.1	80-120	Yes
Calcium	102.9	80-120	Yes
Chromium	99.4	80-120	Yes
Cobalt	94.2	80-120	Yes

Table 8. Laboratory Control Sample (LCS) Results - TCLP Inorganics (continued).

Analyte	LCS Recovery, % (Extraction Fluid #2) ⁴	QC Limits ²	Compliant?
Copper	103.3	80-120	Yes
Iron	102.0	80-120	Yes
Lead	99.0	80-120	Yes
Manganese	97.6	80-120	Yes
Mercury	101 (10/7/99)	80-120	Yes
Nickel	96.6	80-120	Yes
Potassium	104 (10/8/99)	80-120	Yes
Selenium	100.6	80-120	Yes
Silver	104.1	80-120	Yes
Thallium	94.5	80-120	Yes
Titanium	101.3	80-120	Yes
Vanadium	102.8	80-120	Yes
Zinc	183.1, 132.0	80-120	No ⁵

¹ Samples were analyzed 10/8/99 unless otherwise indicated in parentheses.

² QC limits reported by laboratory.

³ Deionized water was used as the extraction fluid.

⁴ Samples were analyzed 10/6/99 unless otherwise indicated in parentheses.

⁵ No action necessary as TCLP zinc was not detected in the affected samples (AC-1-AO-01, AC-1-AO-05, and AC-1-AO-06).

Table 9. Laboratory Control Sample (LCS) Results - SPLP Metals

Analyte	LCS Recovery, %		QC Limits ³	Compliant?	
	1 ¹	2 ²		1	2
Antimony	101.0	103.9	80-120	Yes	Yes
Arsenic	94.2	99.4	80-120	Yes	Yes
Barium	100.2, 137.0	119.9	80-120	No ⁴	Yes
Beryllium	96.1	101.3	80-120	Yes	Yes
Boron	77.8, 123.6	116.7	80-120	No ⁵	Yes
Cadmium	96.4	102.9	80-120	Yes	Yes
Calcium	99.1	103.0	80-120	Yes	Yes
Chromium	99.2	104.8	80-120	Yes	Yes
Chromium ⁶⁺	94.7 (10/1/99)	98.0 (10/1/99)	80-120	Yes	Yes
Cobalt	95.4	101.4	80-120	Yes	Yes
Copper	100.4	101.7	80-120	Yes	Yes
Iron	99.9	101.3	80-120	Yes	Yes
Lead	96.3	102.7	80-120	Yes	Yes
Manganese	98.7	104.3	80-120	Yes	Yes
Mercury	98.5 (10/7/99)	99.9 (10/7/99)	80-120	Yes	Yes
Nickel	96.8	103.5	80-120	Yes	Yes
Potassium	105 (10/4/99)		80-120	Yes	Yes
Selenium	88.1	96.2	80-120	Yes	Yes
Silver	98.9	99.7	80-120	Yes	Yes
Sodium	102 (10/4/99)	106 (10/4/99)	80-120	Yes	Yes
Thallium	93.7	98.7	80-120	Yes	Yes
Titanium	101.7	103.5	80-120	Yes	Yes
Vanadium	99.2	103.5	80-120	Yes	Yes
Zinc	114.7	118.7	80-120	Yes	Yes

¹ Samples were analyzed 10/6/99 unless otherwise indicated in parentheses.

² Samples were analyzed 10/15/99 unless otherwise indicated in parentheses.

³ QC limits reported by laboratory.

⁴ Positive results for SPLP barium in sample AC-1-AO-05 were qualified as estimated with a high bias (K).

⁵ Positive results for SPLP boron in AC-1-AO-05 and AC-1-AO-06 were qualified as estimated (J).

Table 10. Field Duplication Results ¹

EPA Sample Number	AC-1-AO-01	AC-1-AO-05	Relative Percent Difference ²
Sample Description	Reduction furnace slag (Sb < 5%)	Reduction furnace slag (Sb < 5%) - field duplicate	
Target Analyte	Result	Result	
Total inorganics:			
Units:	mg/kg	mg/kg	
Antimony	11500	19600	52.1 ³
Arsenic	301	349	14.8
Barium	294	311	5.6
Calcium	8130	8730	7.1
Copper	52.2	65.4	22.4
Iron	13600	14800	8.5
Lead	135	219	47.5
Manganese	160	174	8.4
Potassium	2020	2310	13.4
Sodium	321000	329000	2.5
Titanium	2440	2650	8.3
Physical Properties			
Specific gravity (unitless)	2.7	2.8	3.6
TCLP inorganics:			
Units:	mg/L	mg/L	
Antimony	55.8	88.4	45.2
Arsenic	2	2.3	14.0
Boron	9.8	15.1	42.6
Calcium	20.1	19.3	4.1
Iron	1.3	1.4	7.4
Nickel	0.6	<0.2	200.0 ⁴
Potassium	89.6	90.5	1.0
Selenium	0.6	0.8	28.6
Titanium	0.067	<0.05	200.0 ⁴
Vanadium	1.3	1.4	7.4
SPLP inorganics:			
Units:	mg/L	mg/L	
Antimony	114	113	0.9

Table 10. Field Duplication Results ¹ (continued).

EPA Sample Number	AC-1-AO-01	AC-1-AO-05	Relative Percent Difference ²
Sample Description	Reduction furnace slag (Sb < 5%)	Reduction furnace slag (Sb < 5%) - field duplicate	
Target Analyte	Result	Result	
Arsenic	2.93	2.75	6.3
Barium	<2	0.135	200.0 ⁴
Beryllium	0.0034	0.0032	6.1
Boron	9.27	9.72	4.7
Calcium	3.42	3.63	6.0
Cobalt	0.0061	0.0072	16.5
Copper	0.0087	0.008	8.4
Iron	0.662	0.866	26.7
Potassium	87.6	85.9	2.0
Selenium	0.55	0.493	10.9
Sodium	13000	13800	6.0
Titanium	<0.005	0.0063	200.0
Vanadium	1.14	1.2	5.1

- ¹ Results are only reported for analytes that were detected in at least one of the samples.
- ² The QC limits are 0-50% relative percent difference for sample results greater than 5 times the reporting limit.
- ³ Positive results for this analyte were estimated in samples AC-1-AO-01, AC-1-AO-05, and AC-1-AO-06.
- ⁴ No action necessary as the sample results were not significantly greater than the reporting limit.

Appendix A
Glossary of Data Qualifier Codes

GLOSSARY OF DATA QUALIFIERS CODES

Code	Definition
Codes Relating to Identification	
(NO CODE)	Confirmed identification.
U	Not detected. The associated number indicates approximate sample concentration necessary to be detected.
B	Detected at greater than the reporting limit but not substantially above the level reported in laboratory or field blanks.
R	Results are rejected. Analyte may or may not be present in the sample. Supporting data necessary to confirm result.
Codes Relating to Quantitation	
J	Analyte present. Reported value may not be accurate or precise. This qualifier is applied in cases where the relative percent difference between duplicate analyses (matrix spike/matrix spike duplicate, laboratory duplicate, and/or field duplicate) is outside the QC limits.
K	Analyte present. Reported value may be biased high. Actual value is expected to be lower. This qualifier is applied in cases where the matrix spike, post-digestion spike, or laboratory control sample recovery is higher than the QC limits.
L	Analyte present. Reported value may be biased low. Actual value is expected to be higher. This qualifier is applied in cases where samples were analyzed outside holding times, or where the matrix spike, post-digestion spike, or laboratory control sample recovery is lower than the QC limits.
UJ	Not detected; reporting limit may be inaccurate or imprecise. This qualifier is applied in cases where the relative percent difference between duplicate analyses (matrix spike/matrix spike duplicate, laboratory duplicate, and/or field duplicate) is outside the QC limits.
UL	Not detected; reporting limit is probably higher. This qualifier is applied in cases where samples were analyzed outside holding times, or where the matrix spike, post-digestion spike, or laboratory control sample recovery is lower than the QC limits.

**Appendix B
List of Acronyms**

List of Acronyms

AA	Atomic Absorption Spectroscopy
AL	Action Level
ASTM	American Society for Testing and Materials
CBI	Confidential Business Information
CCB	Continuing Calibration Blank
CCV	Continuing Calibration Verification
COC	Chain of Custody
CVAA	Cold Vapor Atomic Absorption Spectroscopy
%D	Percent Difference
DOT	Department of Transportation
DQ	Data Qualifier
DQA	Data Quality Assessment
DQO	Data Quality Objectives
EPA	Environmental Protection Agency
GC/MS	Gas Chromatography/Mass Spectrometry
GFAA	Gas Furnace Atomic Absorption Spectroscopy
HRGC	High Resolution Gas Chromatography
HRMS	High Resolution Mass Spectrometry
ICB	Initial Calibration Blank
ICP	Inductively Coupled Argon Plasma Spectroscopy
ICS	Interference Check Sample
ICV	Initial Calibration Verification
IDL	Instrument Detection Limit
IS	Internal Standard
LCS	Laboratory Control Sample
MDL	Method Detection Limit
MS	Matrix Spike
MSD	Matrix Spike Duplicate
PAH	Polynuclear Aromatic Hydrocarbon
PDS	Post-Digestion Spike
QA	Quality Assurance
QA/QC	Quality Assurance/Quality Control
QAPP	Quality Assurance Project Plan
QC	Quality Control
%R	Percent Recovery
RCRA	Resource Conservation and Recovery Act
RPD	Relative Percent Difference
RT	Retention Time
SAP	Sampling and Analysis Plan
SR	Sample result
SA	Spike added
SOP	Standard Operating Procedure
SPLP	Synthetic Precipitation Leaching Procedure
SVOC	Semi-Volatile Organic Compound
TCLP	Toxicity Characteristic Leaching Procedure
VOC	Volatile Organic Compound

**Appendix C
Data Validation Worksheet**

I. DATA COMPLETENESS/REASONABLENESS

A. Completeness: Except as reported below, all required information was included in the data packages, including supporting raw data.

MISSING/INCORRECT INFORMATION	DATE LAB CONTACTED	DATA RECEIVED
Results for total metals in solid samples were corrected for moisture content.	11/1/99	11/1/99 (fax)
Sample results for wet chemistry for AC-1-AO-01 were inadvertently not included in data package.	11/17/99	11/17/99
Case narrative did not contain information about problems with sample analysis due to alkalinity (hex. chrome) or insufficient sample volume.	11/17/99	

Was the sample collection plan outlined in the SAP followed (all samples that were to be collected were collected, all field QC samples to be collected were collected, etc.)?

Yes

B. Reasonableness:

Were results for TCLP and SPLP consistent with total results (in consideration of the minimum 20-fold dilution factor)?

Yes, with the following exception: SPLP sodium in AC-1-AO-02. In this case, the result for SPLP sodium is only slightly greater than what would be expected from a 20-fold dilution of the total sodium result.

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II. HOLDING TIMES Complete table for all samples.

Sample ID	Date Sampled	Analyte	Preparation Date	Analysis Date
AC-1-AO-01	9/23/99	Cr ⁶⁺	10/4/99	10/4/99
		TCLP Cr ⁶⁺	10/6/99	10/8/99
		SPLP Cr ⁶⁺	10/1/99	10/1/99
		Specific gravity	10/12/99	10/12/99
		Moisture	10/1/99	10/2/99
		Metals except mercury	10/1/99	10/15/99 (10/8/99 for Na and K)
		Mercury	10/8/99	10/8/99
		TCLP extraction	—	10/1/99
		SPLP extraction	—	9/29/99, 10/10/99
		TCLP metals except mercury	10/4/99	10/6/99 (10/8/99 for K)
		TCLP mercury	10/6/99	10/7/99
		SPLP metals except mercury	10/14/99	10/15/99 (10/4/99 for Na and K)
		SPLP mercury	10/5/99	10/7/99
		AC-1-AO-02	9/23/99	Cr ⁶⁺
TCLP Cr ⁶⁺	10/6/99			10/8/99
SPLP Cr ⁶⁺	10/1/99			10/1/99
Specific gravity	10/12/99			10/12/99
Moisture	10/1/99			10/2/99
Metals except mercury	10/1/99			10/15/99 (10/8/99 for Na and K)
Mercury	10/8/99			10/8/99
TCLP extraction	—			10/5/99
SPLP extraction	—			9/29/99, 10/10/99
TCLP metals except mercury	10/6/99			10/8/99
TCLP mercury	10/7/99			10/7/99
SPLP metals except mercury	10/14/99			10/15/99 (10/4/99 for Na and K)
SPLP mercury	10/5/99			10/7/99

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Sample ID	Date Sampled	Analyte	Preparation Date	Analysis Date
AC-1-AO-03	9/23/99	Cr ⁶⁺	10/4/99	10/4/99
		TCLP Cr ⁶⁺	10/6/99	10/8/99
		SPLP Cr ⁶⁺	10/1/99	10/1/99
		Specific gravity	10/11/99	10/11/99
		Moisture	10/1/99	10/2/99
		Metals except mercury	10/1/99	10/15/99 (10/8/99 for Na and K)
		Mercury	10/8/99	10/8/99
		TCLP extraction	—	10/5/99
		SPLP extraction	—	9/29/99, 10/10/99
		TCLP metals except mercury	10/6/99	10/8/99
		TCLP mercury	10/7/99	10/7/99
		SPLP metals except mercury	10/14/99	10/15/99 (10/4/99 for Na and K)
		SPLP mercury	10/5/99	10/7/99
		AC-1-AO-05	9/23/99	Cr ⁶⁺
TCLP Cr ⁶⁺	10/6/99			10/8/99
SPLP Cr ⁶⁺	10/1/99			10/1/99
Specific gravity	10/12/99			10/12/99
Moisture	10/1/99			10/2/99
Metals except mercury	10/1/99			10/15/99 (10/8/99 for Na and K)
Mercury	10/8/99			10/8/99
TCLP extraction	—			10/1/99
SPLP extraction	—			9/29/99
TCLP metals except mercury	10/4/99			10/6/99 (10/8/99 for K)
TCLP mercury	10/6/99			10/7/99
SPLP metals except mercury	10/1/99			10/6/99 (10/4/99 for Na and K)
SPLP mercury	10/5/99			10/7/99

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Sample ID	Date Sampled	Analyte	Preparation Date	Analysis Date
AC-1-AO-06	9/23/99	Cr ⁶⁺	10/4/99	10/4/99
		TCLP Cr ⁶⁺	10/6/99	10/8/99
		SPLP Cr ⁶⁺	10/1/99	10/1/99
		Specific gravity	10/12/99	10/12/99
		Moisture	10/1/99	10/2/99
		Metals except mercury	10/1/99	10/15/99 (10/8/99 for Na and K)
		Mercury	10/8/99	10/8/99
		TCLP extraction	—	10/1/99
		SPLP extraction	—	9/29/99, 10/10/99
		TCLP metals except mercury	10/4/99	10/6/99 (10/8/99 for K)
		TCLP mercury	10/6/99	10/7/99
		SPLP metals except mercury	10/1/99	10/6/99 (10/4/99 for Na and K)
		SPLP mercury	10/5/99	10/7/99
		AC-1-AO-07	9/23/99	Cr ⁶⁺
TCLP Cr ⁶⁺	10/6/99			10/8/99
SPLP Cr ⁶⁺	10/1/99			10/1/99
Specific gravity	10/11/99			10/11/99
Moisture	10/1/99			10/2/99
Metals except mercury	10/1/99			10/15/99 (10/8/99 for Na and K)
Mercury	10/8/99			10/8/99
TCLP extraction	—			10/5/99
SPLP extraction	—			9/29/99, 10/10/99
TCLP metals except mercury	10/6/99			10/8/99
TCLP mercury	10/7/99			10/7/99
SPLP metals except mercury	10/14/99			10/15/99 (10/4/99 for Na and K)
SPLP mercury	10/5/99			10/7/99
AC-1-AO-04	9/23/99			Cr ⁶⁺
		Specific gravity	10/11/99	10/11/99
		pH	10/1/99	10/1/99
		Oxidation/reduction	10/13/99	10/13/99
		Metals except mercury	10/7/99	10/8/99 (10/11/99 for K and 10/12/99 for Na)
		Mercury	10/8/99	10/8/99

Metals - 180 days from sample collection
Mercury - 28 days from sample collection

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- TCLP & SPLP - 180 days from sample collection to extraction and 180 days from extraction to analysis for total metals
- 28 days from sample collection to extraction for mercury
- Chromium⁶⁺ - one month from sample collection to extraction and 4 days from extraction to analysis for solid samples
- 24 hours from sample collection to analysis for aqueous samples
- Nitrite/Nitrate - 48 hours from sample collection
- Ammonia - 28 days from sample collection
- CN - 14 days from sample collection
- TOC - 28 days from sample collection
- TSS - 7 days from sample collection

ACTIONS: Holding time for Cr⁶⁺ was exceeded in sample AC-1-AO-04; sample results were qualified as estimated with a low bias (L(+);UL(ND)).

NOTE: Extraction fluid #1 was used for TCLP metals extraction of AC-1-AO-02, AC-1-AO-03, and AC-1-AO-07, and extraction fluid #2 was used for TCLP metals extraction of AC-1-AO-01, AC-1-AO-05, and AC-1-AO-06. Extraction fluid #1 was used for SPLP metal extractions.

- Action: 1. If holding times are exceeded all positive results are estimated with a low bias (L) and non-detects are estimated with a low bias (UL).
- 2. If holding times are grossly exceeded (>2x), all results are qualified unusable (R).

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1. Recovery Criteria

List the analytes which did not meet the percent recovery (%R) criteria for Initial or Continuing Calibration.

ANALYSIS	ICV/CCV#	ANALYTE	%R	ACTION	SAMPLES AFFECTED

ACTIONS: None; all criteria met.

If any analyte does not meet the %R criteria follow the actions stated below:

Positive Results	Non-detected Results	%R	
		Methods 6010 and 7196	Methods 7470, 7471, 7610, and 7770
L	UL	<90	<80
K		>110	>120

$$\text{Recovery (\%)} = \frac{\text{QC Result}}{\text{True Value}} \times 100$$

2. Analytical Sequence

- A. Did the laboratory use the proper number of standards for calibration as described in method? Yes
- B. Were calibrations performed at the beginning of each analysis? Yes
- C. Were calibration standards analyzed at the beginning of sample analysis and at a minimum frequency of ten percent or every two hours during analysis, whichever is more frequent? Yes
- D. Were the correlation coefficients for the calibration curves for Hg, nitrate, nitrite, ammonia, sodium, potassium, TOC, hexavalent chromium, and CN ≥ 0.995 ? Yes

ACTION: None; all criteria met.

1. If the minimum number of standards were not used for initial calibration or if the instrument was not calibrated daily and each time the instrument was set up, reject the associated data (R).
2. If the correlation coefficient is <0.995 , qualify results $>IDL$ as estimated (J), and results $<IDL$ as estimated (UJ).

List the blank contamination in Section 1 below.

1. Laboratory Blanks

ANALYSIS: Total

DATE	ICB/CCB#	PREP BL	ANALYTE	CONC/UNITS

ANALYSIS: TCLP

DATE	ICB/CCB#	PREP BL	ANALYTE	CONC/UNITS

ANALYSIS: SPLP

DATE	ICB/CCB#	PREP BL	ANALYTE	CONC/UNITS

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List the blank contamination in Section 2 below.

2. Equipment/Trip Blanks

SAMPLE NUMBER	ANALYTE	CONCENTRATION (mg/L)
AC-1-AO-04	antimony	0.0371
	calcium	0.235

3. Frequency Requirements

- A. Was a preparation blank analyzed for each matrix, for every 20 samples and for each digestion batch? Yes
 - B. Was a calibration blank run every 10 samples or every 2 hours whichever is more frequent (inorganics analyses only)? Yes
 - C. Were the ICB/CCB results <3xIDL (Method 6010 only)? Yes
- If not, were samples reanalyzed? _____

If no, the data may be affected. Use professional judgement to determine the severity of the effect and qualify the data accordingly. Discuss any actions below and list the samples affected.

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IV. BLANK ANALYSIS RESULTS (Section 4)

4. Blank Actions

The Action Level for any analyte is equal to five times the highest concentration of that element's contamination in any blank. The action level for samples which have been concentrated or diluted should be multiplied by the concentration/dilution factor. No positive sample results should be reported unless the concentration of the analyte in the sample exceeds the Action Level (AL). Specific actions are as follows:

1. When the concentration is greater than the IDL, but less than the Action Level, report the sample concentration detected with a B.
2. When the sample concentration is greater than the Action Level, report the sample concentration unqualified.

ANALYTE	MAX. CONC. (mg/L)	AL (mg/L)	ANALYTE	MAX. CONC./UNITS	AL/UNITS
Total antimony	0.0371	0.1855			
Total calcium	0.235	1.175			

ACTION: No qualification of data necessary as sample results for the affected analytes either greatly exceeded the action levels or were non-detected.

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V. ICP INTERFERENCE CHECK SAMPLE (Section 1 & 2)

1. Recovery Criteria

List any elements in the ICS AB solution which did not meet the criteria for %R.

DATE	ELEMENT	%R	ACTION	SAMPLES AFFECTED

$$Recovery (\%) = \frac{QC\ Result}{True\ Value} \times 100$$

ACTIONS: None; all criteria met.

For samples with concentrations of Al, Ca, Mg, and Fe or other potential interferents which are 50% or more than their respective levels in the ICS, the following actions apply if an element does not meet the %R criteria:

%R	Positive results	Non-detected results
<50%	R	R
50-79%	L	UL
>120%	K	

2. Frequency Requirements

Were Interference QC samples run at the beginning of each sample analysis run? Yes

If no, the data may be affected. Use professional judgement to determine the severity of the effect and qualify the data accordingly. Discuss any actions below and list the samples affected.

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Sample No.: AC-1-AO-01

Analysis: Total

1. Recovery Criteria

List the percent recoveries for analytes which did not meet the required criteria.

$$\text{Recovery (\%)} = \frac{(\text{SSR} - \text{SR})}{\text{SA}} \times 100$$

where SSR = Spiked sample result
 SR = Sample result
 SA = Spike added

ANALYTE	SAMPLE	SSR	SR	SA	%R	ACTION
Sb	MS	12468.3467	11478.0479	50	1980.6	None; see 1 below
	MSD	20054.9258			17153.8	
As	MS	375.6828	300.5543	50	150.3	None; see 1 below
	MSD	392.2986			183.5	
Ba	MSD	329.8440	293.5864	50	72.5	None; see 1 below
B	MS	277.0195	202.7996	50	148.4	None; see 1 below
Cu	MSD	132.6556	52.2124	50	160.9	K(+)
Fe	MS	14477.8545	13577.6768	200	450.1	None; see 1 below
	MSD	14230.8184			326.6	
Pb	MS	289.6072	135.3020	50	308.6	J(+)
	MSD	353.6293			436.7	
Mn	MS	223.5038	160.0428	50	126.9	K(+)
Ag	MSD	28.6195	3.4301	20	125.9	K(+)
Na	MS	332600	321100	5000	230	None; see 1 below
	MSD	317700			-68	
Ti	MS	2607.0579	2439.2412	50	335.6	None; see 1 below
	MSD	2557.3684			236.3	
Zn	MS	471.7370	284.7828	50	373.9	None; see 1 below
	MSD	412.0526			254.5	

Matrix Spike Actions apply to all samples of the same matrix.

ACTIONS: See table above. Actions only apply to samples AC-1-AO-01, AC-1-AO-05, and AC-1-AO-06. For lead, the use of the J qualifier was based on the results of duplicate sample analysis (not reported here) which had a relative percent difference of 99.4%.

1. If the sample concentration exceeds the spike concentration by a factor of 4 or more, no action is taken.
2. If any analyte does not meet the %R criteria, follow the actions stated below:

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Positive Results	Non-detected Results	%R	
		Metals/CN	Ammonia/Nitrate/Nitrite
L/R ¹	R	<50%	<50%
L	UL	50-74%	50-79%
K		>125%	>120
K/R ¹		>135%	>135

¹ Professional judgement will be used to determine the appropriate data qualifier.

2. Frequency Criteria

A. Was a matrix spike prepared at the required frequency? Yes

A separate worksheet should be used for each matrix spike pair.

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Sample No.: AC-1-AO-01

Analysis: TCLP

1. Recovery Criteria

List the percent recoveries for analytes which did not meet the required criteria.

$$Recovery (\%) = \frac{(SSR - SR)}{SA} \times 100$$

where SSR = Spiked sample result
 SR = Sample result
 SA = Spike added

ANALYTE	SAMPLE	SSR	SR	SA	%R	ACTION
B	MS	20.5913	9.7790	5	216.2	K(+)

Matrix Spike Actions apply to all samples of the same matrix.

ACTIONS: See table above. Actions apply only to samples AC-1-AO-01, AC-1-AO-05, and AC-1-AO-06.

- If the sample concentration exceeds the spike concentration by a factor of 4 or more, no action is taken.
- If any analyte does not meet the %R criteria, follow the actions stated below:

Positive Results	Non-detected Results	%R	
		Metals/CN	Ammonia/Nitrate/Nitrite
L/R ¹	R	<50%	<50%
L	UL	50-74%	50-79%
K		>125%	>120
K/R ¹		>135%	>135

¹ Professional judgement will be used to determine the appropriate data qualifier.

2. Frequency Criteria

A. Was a matrix spike prepared at the required frequency? Yes

A separate worksheet should be used for each matrix spike pair.

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Sample No.: AC-1-AO-01

Analysis: SPLP

1. Recovery Criteria

List the percent recoveries for analytes which did not meet the required criteria.

$$\text{Recovery (\%)} = \frac{(\text{SSR} - \text{SR})}{\text{SA}} \times 100$$

where SSR = Spiked sample result
 SR = Sample result
 SA = Spike added

ANALYTE	SAMPLE	SSR	SR	SA	%R	ACTION
Sb	MS	92814.9844	114164.9	250	-8540	None; see 1 below
	MSD	92886.7031			-8511.3	
As	MS	2793.3965	2933.7000	250	-56.1	None; see 1 below
	MSD	2781.7578			-60.8	
Be	MS	38.9926	3.3808	50	71.2	L(+);UL(ND)
	MSD	38.8804			71.0	
B	MS	8035.9453	9270.2530	250	-493.7	None; see 1 below
	MSD	8027.8247			-497.0	
Cd	MS	34.9702	ND	50	69.9	L(+);UL(ND)
	MSD	35.4386			70.9	
Fe	MS	1334.9432	661.9139	1000	67.3	L(+);UL(ND)
	MSD	1340.5002			67.9	
Ni	MS	179.6158	2.0723	250	71.8	L(+);UL(ND)
	MSD	179.8083			71.9	
Na	MS	13780	12960	25	3280	None; see 1 below
	MSD	13670			2840	
Tl	MS	182.5332	ND	250	73.0	L(+);UL(ND)
	MSD	183.4025			73.4	
Ti	MS	185.1014	4.5319	250	74.0	L(+);UL(ND)
	MSD	180.8244			72.3	
V	MS	1319.6436	1135.3738	250	73.7	None; see 1 below
	MSD	1312.3638			70.8	

Matrix Spike Actions apply to all samples of the same matrix.

ACTIONS: See table above. Actions apply only to samples AC-1-AO-01, AC-1-AO-05, AC-1-AO-06.

- If the sample concentration exceeds the spike concentration by a factor of 4 or more, no action is taken.

Data Review Worksheets

2. If any analyte does not meet the %R criteria, follow the actions stated below:

Positive Results	Non-detected Results	% R	
		Metals/CN	Ammonia/Nitrate/Nitrite
L/R ¹	R	<50%	<50%
L	UL	50-74%	50-79%
K		>125%	>120
K/R ¹		>135%	>135

¹ Professional judgement will be used to determine the appropriate data qualifier.

2. Frequency Criteria

A. Was a matrix spike prepared at the required frequency? Yes

A separate worksheet should be used for each matrix spike pair.

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3. Duplicate Criteria

Sample No.: AC-1-AO-01

Analysis: Total

List the relative percent difference (RPD) for analytes which did not meet the required criteria.

$$RPD (\%) = \frac{|MS-MSD|}{(MS+MSD)/2} \times 100$$

where RPD = Relative percent difference
 MS = Matrix spike value
 MSD = Matrix spike duplicate value

ANALYTE	MS	MSD	RPD	ACTION
Sb	12468.3467	20054.9258	46.7	J(+)

Matrix Spike Actions apply to all samples of the same matrix.

ACTIONS: See table above. Actions apply only to AC-1-AO-01, AC-1-AO-05, AC-1-AO-06. The laboratory also conducted duplicate analyses of AC-1-AO-01 for moisture, and duplicate analyses of AC-1-AO-04 for pH; all results were within criteria.

- For positive results which have an RPD >25%, use professional judgement to determine whether to estimate (J) or reject (R) the results.

A separate worksheet should be used for each matrix spike pair.

US EPA ARCHIVE DOCUMENT

3. Duplicate Criteria

Sample No.: AC-1-AO-01

Analysis: TCLP

List the relative percent difference (RPD) for analytes which did not meet the required criteria.

$$RPD (\%) = \frac{|MS-MSD|}{(MS+MSD)/2} \times 100$$

where RPD = Relative percent difference
 MS = Matrix spike value
 MSD = Matrix spike duplicate value

ANALYTE	MS	MSD	RPD	ACTION
B	20.5913	15.1617	30.4	J(+)

Matrix Spike Actions apply to all samples of the same matrix.

ACTIONS: See table above. Actions apply only to AC-1-AO-01, AC-1-AO-05, AC-1-AO-06.

- For positive results which have an RPD >25%, use professional judgement to determine whether to estimate (J) or reject (R) the results.

A separate worksheet should be used for each matrix spike pair.

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3. Duplicate Criteria

Sample No.: AC-1-AO-01

Analysis: SPLP

List the relative percent difference (RPD) for analytes which did not meet the required criteria.

$$RPD (\%) = \frac{|MS-MSD|}{(MS+MSD)/2} \times 100$$

where RPD = Relative percent difference
 MS = Matrix spike value
 MSD = Matrix spike duplicate value

ANALYTE	MS	MSD	RPD	ACTION

Matrix Spike Actions apply to all samples of the same matrix.

ACTIONS: None; all criteria met.

- For positive results which have an RPD >25%, use professional judgement to determine whether to estimate (J) or reject (R) the results.

A separate worksheet should be used for each matrix spike pair.

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Sample ID AC-1-AO-01

Analysis: Total

- List the percent recoveries for analytes which did not meet the required criteria in the post-digestion spike sample.

S - amount of spike added
 SSR - spiked sample result
 SR - sample result

SAMPLE	ANALYTE	SSR	SR	S	%R	ACTION

Post-Digestion Spike Actions apply to all samples of the same matrix.

ACTIONS: Although the laboratory conducted post-digestion spike analyses for all ICP analytes, post-digestion spike results were only reported for analytes for which matrix spike and matrix spike duplicate recovery failed. No post-digestion spike results were reported for this sample.

- If the sample concentration exceeds the spike concentration by a factor of 4 or more, no action is taken.
- If any analyte does not meet the %R criteria, follow the actions stated below:

Positive results	Non-detected results	%R	
		Methods 6010 and 7196	Methods 7470, 7471, 7740, and 7841
L	R	<30%	<30%
L	UL	30-74%	30-84%
K		>125%	>115%

- Frequency Criteria

Was a post-digestion spike prepared at the required frequency? Yes

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Sample ID AC-1-AO-01

Analysis: TCLP

- List the percent recoveries for analytes which did not meet the required criteria in the post-digestion spike sample.

S - amount of spike added
 SSR - spiked sample result
 SR - sample result

SAMPLE	ANALYTE	SSR	SR	S	%R	ACTION

Post-Digestion Spike Actions apply to all samples of the same matrix.

ACTIONS: Although the laboratory conducted post-digestion spike analyses for all ICP analytes, post-digestion spike results were only reported for analytes for which matrix spike and matrix spike duplicate recovery failed. No post-digestion spike results were reported for this sample.

- If the sample concentration exceeds the spike concentration by a factor of 4 or more, no action is taken.
- If any analyte does not meet the %R criteria, follow the actions stated below:

Positive results	Non-detected results	%R	
		Methods 6010 and 7196	Methods 7470, 7471, 7740, and 7841
L	R	<30%	<30%
L	UL	30-74%	30-84%
K		>125%	>115%

- Frequency Criteria

Was a post-digestion spike prepared at the required frequency? Yes

Sample ID AC-1-AO-01

Analysis: SPLP

- List the percent recoveries for analytes which did not meet the required criteria in the post-digestion spike sample.

S - amount of spike added
 SSR - spiked sample result
 SR - sample result

ANALYTE	SSR	SR	S	%R	ACTION
Be	40.2021	3.3808	50	73.6	L(+);UL(ND)
Cd	36.9357	ND	50	73.9	L(+);UL(ND)
Fe	1361.1605	661.9139	1000	69.9	L(+);UL(ND)
Ni	182.1548	2.0723	250	72.9	L(+);UL(ND)

Post-Digestion Spike Actions apply to all samples of the same matrix.

ACTIONS: See table above. Although the laboratory conducted post-digestion spike analyses for all ICP analytes, post-digestion spike results were only reported for analytes for which matrix spike and matrix spike duplicate recovery failed. Actions apply only to AC-1-AO-01, AC-1-AO-05, AC-1-AO-06.

- If the sample concentration exceeds the spike concentration by a factor of 4 or more, no action is taken.
- If any analyte does not meet the %R criteria, follow the actions stated below:

Positive results	Non-detected results	%R	
		Methods 6010 and 7196	Methods 7470, 7471, 7740, and 7841
L	R	<30%	<30%
L	UL	30-74%	30-84%
K		>125%	>115%

- Frequency Criteria

Was a post-digestion spike prepared at the required frequency? Yes

Sample Nos.: AC-1-AO-01, AC-1-AO-05

Matrix: solid

List the concentrations of the compounds for which RPD is $\geq 50\%$.

ANALYTE	SAMPLE CONC.	DUP SAMPLE CONC.	RPD	ACTION
Total antimony	11500	19600	52.1	J(+)
TCLP nickel	0.6	<0.2	200.0	None; <5xRL
TCLP titanium	0.067	<0.05	200.0	None; <5xRL
SPLP barium	<2	0.135	200.0	None; <5xRL
SPLP titanium	<0.005	0.0063	200.0	None; <5xRL

NOTE: Professional judgement may be utilized to apply duplicate actions.

A separate worksheet should be filled out for each field duplicate pair.

ACTIONS: See table above. Actions apply only to AC-1-AO-01, AC-1-AO-05, AC-1-AO-06.

1. Estimate (J) positive results for elements which have an RPD $\geq 50\%$ when sample results are $\geq 5x$ the reporting limit.
2. If sample results are less than $5x$ the reporting limit, estimate (J) positive results for elements whose absolute difference is $\geq 4x$ the reporting limit. If both samples are non-detected, the RPD is not calculated (NC).

1. Aqueous LCS

List any LCS recoveries not within the 80-120% criteria and the samples affected.

DATE	ANALYTE	%R	ACTION	SAMPLES AFFECTED
10/6/99	TCLP zinc (extraction fluid #2)	132.0, 183.1	K(+)	AC-1-AO-01, AC-1-AO-05, AC-1-AO-06 (TCLP)
10/6/99	SPLP barium	137.0	K(+)	AC-1-AO-05 and AC-1-AO-06 (SPLP)
10/6/99	SPLP boron	77.8, 123.6	J(+);UJ(ND)	

2. Solid LCS

List any analytes that were not within the control windows set for the solid LCS sample. The 80-120% criteria is not used evaluate solid LCS results.

ELEMENT	DATE	LCS %R	CONTROL WINDOWS	ACTION	SAMPLES AFFECTED

ACTIONS: See table above.

Aqueous LCS:

%R	Positive results	Non-detected results
<50%	R	R
50-79%	L	UL
>120%	K	

Solid LCS:

	Positive results	Non-detected results
<Control Windows	L	UL
>Control Windows	K	

3. Frequency Criteria

A. Was an LCS analyzed for every matrix, every digestion batch, and every 20 samples?

Yes

Data Review Worksheets

X. ICP SERIAL DILUTION ANALYSIS

_____ Serial dilutions were performed for each matrix and results of the diluted sample analysis agreed within ten percent of the original undiluted analysis for analyte concentrations greater than 50x the LOD before dilution.

_____ Serial dilutions were not performed for the following: _____

_____ Serial dilutions were performed, but analytical results did not agree within 10% for analyte concentrations greater than 50x the LOD before dilution.

Report all results below that do not meet the required laboratory criteria for ICP serial dilution analysis.

Analysis: Total

ELEMENT	LOD	50xLOD	SAMPLE RESULT	SERIAL DILUTION	%D	ACTION

Actions apply to all samples of the same matrix.

ACTIONS: The laboratory conducted serial dilution analyses for ICP analytes only in cases for which matrix spike recovery failed, the sample result was greater than 4 times the spike added, and the sample result was greater than 50 times the reporting limit. For this sample, serial dilution was required for antimony, arsenic, barium, iron, titanium, and zinc. However, the sample was run at dilutions of 1:100 and 1:1000 to get these sample results within calibration range.

1. Estimate (J) positive results if %D >10%.

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_____ Serial dilutions were performed for each matrix and results of the diluted sample analysis agreed within ten percent of the original undiluted analysis for analyte concentrations greater than 50x the LOD before dilution.

_____ Serial dilutions were not performed for the following: _____

_____ Serial dilutions were performed, but analytical results did not agree within 10% for analyte concentrations greater than 50x the LOD before dilution.

Report all results below that do not meet the required laboratory criteria for ICP serial dilution analysis.

Analysis: TCLP

ELEMENT	LOD	50xLOD	SAMPLE RESULT	SERIAL DILUTION	%D	ACTION

Actions apply to all samples of the same matrix.

ACTIONS: The laboratory conducted serial dilution analyses for ICP analytes only in cases for which matrix spike recovery failed, the sample result was greater than 4 times the spike added, and the sample result was greater than 50 times the reporting limit. For this sample, no serial dilutions were required.

1. Estimate (J) positive results if %D >10%.

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_____ Serial dilutions were performed for each matrix and results of the diluted sample analysis agreed within ten percent of the original undiluted analysis for analyte concentrations greater than 50x the LOD before dilution.

_____ Serial dilutions were not performed for the following: _____

_____ Serial dilutions were performed, but analytical results did not agree within 10% for analyte concentrations greater than 50x the LOD before dilution.

Report all results below that do not meet the required laboratory criteria for ICP serial dilution analysis.

Analysis: SPLP

ELEMENT	LOD	50xLOD	SAMPLE RESULT	SERIAL DILUTION	%D	ACTION

Actions apply to all samples of the same matrix.

ACTIONS: The laboratory conducted serial dilution analyses for ICP analytes only in cases for which matrix spike recovery failed, the sample result was greater than 4 times the spike added, and the sample result was greater than 50 times the reporting limit. For this sample, serial dilution was required for antimony, arsenic, boron, and vanadium. However, the sample was run at dilutions of 1:10 and 1:100 to get these sample results within calibration range.

1. Estimate (J) positive results if %D >10%.

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1. Instrument Detection Limits (IDLs)

_____ IDL results were present and found to be less than the required detection limits.

_____ IDLs were not included in the data package.

X IDLs were present, but the criteria was not met for the following elements: arsenic in wastewaters and leachates (0.00237 mg/L versus 0.002 mg/L target) and thallium in wastewaters and leachates (0.00216 mg/L versus 0.0005 mg/L target).

Detection limit requirements were not specified for familiarization samples.

2. Reporting Requirements

Yes Were sample results on Form 1s reported down to the PQL for all analytes?

Yes Were sample weights, volumes, and dilutions taken into account when reporting detection limits?

If not, the reported results may be inaccurate. Make the necessary changes on the data summary tables and request that the laboratory resubmit the corrected data.

NOTE: Several samples had elevated detection limits due to dilutions required to bring results for some analytes within the calibration range of the instrument, and because of high levels of sodium in the samples (which clogged the instrument).

Data Review Worksheets

XII. SAMPLE QUANTITATION

X Samples results fall within the linear range for ICP and within the calibrated range for all other parameters.

_____ Samples results were beyond the linear range/calibration range of the instrument for the following samples/elements: _____

In the space below, please show a minimum of one sample calculation per method type:

ICP: Cr in AC-1-AO-01 (total): results were reported directly in mg/kg using a correction factor, 10, calculated as follows:

$$\text{results in ug/L} \times \frac{100 \text{ mL}}{1.00 \text{ g}} \times \frac{1 \text{ L}}{1000 \text{ mL}} \times \frac{1000 \text{ g}}{1 \text{ kg}} \times \frac{1 \text{ mg}}{1000 \text{ ug}} \times 100 \text{ (dilution factor)} = \text{results in mg/kg}$$

Flame AA: Na in AC-1-AO-01 (total):

$$3211 \text{ mg/L} \times \frac{100 \text{ mL}}{1.00 \text{ g}} \times \frac{1 \text{ L}}{1000 \text{ mL}} \times \frac{1000 \text{ g}}{1 \text{ kg}} = 321100 \text{ mg/kg}$$

CVAA: Hg in AC-1-AO-02 (total):

$$1.704 \text{ ug/L} \times \frac{96 \text{ mL}}{0.6 \text{ g}} \times \frac{1 \text{ L}}{1000 \text{ mL}} \times \frac{1000 \text{ g}}{1 \text{ kg}} \times \frac{1 \text{ mg}}{1000 \text{ ug}} = 0.273 \text{ mg/kg}$$

Colorimetric: Cr⁶⁺ in AC-1-AO-02:

$$-0.0012 \text{ mg/L} \times \frac{50 \text{ mL}}{50.00 \text{ g}} \times \frac{1 \text{ L}}{1000 \text{ mL}} \times \frac{1000 \text{ g}}{1 \text{ kg}} = -0.0012 \text{ mg/kg} (< 0.02 \text{ mg/kg})$$

For soil samples, the following equation may be necessary to convert raw data values (usually reported in : g/L) to actual sample concentrations (mg/kg):

$$\text{Conc. in ug/L} \times \frac{\text{volume diluted to (mL)}}{\text{weight digested (g)}} \times \frac{1 \text{ L}}{1000 \text{ mL}} \times \frac{1000 \text{ g}}{1 \text{ kg}} \times \frac{1 \text{ mg}}{1000 \text{ ug}} = \text{mg/kg}$$

**Appendix D
Sample Volumes Collected**

Table 1 - Samples Collected

Sample Sample Number	Container Type and Size	Number of Containers ¹	Analysis	Preservation
Reduction Furnace Slag (Sb<5%) AC-1-AO-01 AC-1-AO-01-MS AC-1-AO-01-MSD (RIN 3)	8 oz wide mouth jar with Teflon-lined cap	12	Total Metals	# 4 °C
			TCLP - Metals, Cr ⁺⁶	# 4 °C
			SPLP - Metals, Cr ⁺⁶	# 4 °C
			Cr ⁺⁶	# 4 °C
			specific gravity, moisture content	None
Reduction Furnace Slag (5%<Sb<10%) AC-1-AO-06 (RIN 3)	8 oz wide mouth jar with Teflon-lined cap	4	Total Metals	# 4 °C
			TCLP - Metals, Cr ⁺⁶	# 4 °C
			SPLP - Metals, Cr ⁺⁶	# 4 °C
			Cr ⁺⁶	# 4 °C
			specific gravity, moisture content	None
Furnace Refractory AC-1-AO-02 (RIN 4)	8 oz wide mouth jar with Teflon-lined cap	4	Total Metals	# 4 °C
			TCLP - Metals, Cr ⁺⁶	# 4 °C
			SPLP - Metals, Cr ⁺⁶	# 4 °C
			Cr ⁺⁶	# 4 °C
			specific gravity, moisture content	None
Oxidation Furnace Baghouse Filters AC-1-AO-03 (RIN 5)	Zip-lock plastic bag 400 gms	8	Total Metals	# 4 °C
			TCLP - Metals, Cr ⁺⁶	# 4 °C
			SPLP - Metals, Cr ⁺⁶	# 4 °C
			Cr ⁺⁶	# 4 °C
			specific gravity, moisture content	None
Reduction Furnace Baghouse Filters AC-1-AO-07 (RIN 5)	Zip-lock plastic bag 400 gms	8	Total Metals	# 4 °C
			TCLP - Metals, Cr ⁺⁶	# 4 °C
			SPLP - Metals, Cr ⁺⁶	# 4 °C
			Cr ⁺⁶	# 4 °C
			specific gravity, moisture content	None

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Sample Sample Number	Container Type and Size	Number of Containers ¹	Analysis	Preservation
Equipment Blank AC-1-AO-04	1 liter plastic with Teflon-lined cap	2	Total Metals	pH<2 using HNO ₃ , # 4 °C
		2	Cr ⁺⁶	# 4 °C
			pH, Eh, specific gravity	None
Field Duplicate AC-1-AO-05 (RIN 3)	8 oz wide mouth jar with Teflon-lined cap	4	Total Metals	# 4 °C
			TCLP - Metals, Cr ⁺⁶	# 4 °C
			SPLP - Metals, Cr ⁺⁶	# 4 °C
			Cr ⁺⁶	# 4 °C
			specific gravity, moisture content	None

1. The number of containers shown includes the number of split sample containers to be collected for the U.S. Antimony Corporation.

Appendix E
Target Analyte List and Detection Limits

Target Analytes and Target Detection Limits. ²

Constituent	Wastewater Leachate Detection Limits, mg/L [Health-Based Drinking Water Level - MCLG] ²	Total Concentration Detection Limits, mg/kg [Method Detection Limits] ³
Inorganics		
Antimony	0.006	2.1
Arsenic	0.001	0.1
Barium	2	0.087
Beryllium	0.004	0.018
Boron	0.0038	0.38
Cadmium	0.005	0.23
Chromium (total)	0.1	0.47
Chromium, hexavalent	0.1	0.012
Cobalt	0.0047	0.47
Copper	1.3	0.36
Lead	0.015	2.8
Manganese	0.05	5
Mercury	0.0005	0.02
Nickel	0.1 ⁴	1
Selenium	0.05	5
Silver	0.0047	0.47
Thallium	0.0005	2.7
Titanium	0.005	0.5
Vanadium	0.005	0.5
Zinc	0.0012	0.12
Physical Properties		
pH ⁵	N/A	N/A
Specific gravity	N/A	N/A
Oxidation/reduction potential	N/A	N/A
Percent moisture	N/A	N/A

1. In all cases in which the target detection limit specified in this table is not achievable for an analyte, the laboratory QA officer will immediately inform the Dynamac Laboratory Coordinator who will promptly inform the EPA WAM.

2. MCLG = Maximum Contaminant Level Goal; MCL = Maximum Contaminant Level. Shaded blocks indicate that no MCLG or MCL exists for these analytes. In these instances, the target detection limit is the estimated detection limit of the method specified in Table 2-1.

3. The Superfund soil screening levels (ingestion) were the health-based criteria used to determine target detection limits in this column. However, because the estimated detection limits (for the methods specified in Table 2-1) were below the soil screening levels the method detection limit was chosen as the target detection limit for each analyte.

4. The MCLG and MCL for nickel are being remanded.
5. Measure and document the pH of all wastes, and TCLP and SPLP leachates.

Appendix F
Preparation and Analytical Methods for
Analysis of U.S. Antimony Corporation Antimony Oxide Residuals

Analysis	Matrix	Preparation Method ¹	Analytical Method ¹	Sample Container ²	Preservative	Holding Time
Metals, except mercury ³	Wastewater	3010A	6010B	P, G	Adjust to pH<2 using HNO ₃	6 months
	Sludge/Solid waste	3050B			—	
Arsenic ⁴	Wastewater	7060A	7060A	P, G	Adjust to pH<2 using HNO ₃	6 months
	Sludge/Solid waste	3050B			—	
Mercury	Wastewater	7470A	7470A	P, G	Adjust to pH<2 using HNO ₃	28 days
	Sludge/Solid waste	7471A	7471A		Cool (4 °C)	
Hexavalent chromium	Wastewater	—	7196A	P, G	Cool (4 °C)	24 hours
	Sludge/Solid waste	3060A				1 month until extraction; 4 days from extraction to analysis
TCLP Metals, except mercury ^{6,7}	All matrices	1311	6010B	P, G	Cool (4 °C); No preservative	180 days ⁸
TCLP Mercury			7470A			28 days ⁸
TCLP hexavalent chromium			7196A			1 month until extraction; 24 hours from extraction to analysis
SPLP Metals, except mercury ^{6,7}	All matrices	1312 ¹⁰	6010B	P, G	Cool (4 °C); No preservative	180 days ¹¹
SPLP Mercury			7470A			28 days ¹¹
SPLP hexavalent chromium			7196A			1 month until extraction; 24 hours from extraction to analysis
pH	Wastewater	—	9040B	P, G	—	Analyze as soon as possible
Eh	All matrices	—	ASTM D1498-93	P, G	—	Analyze as soon as possible
Specific Gravity	All matrices	—	ASTM D 2937	P, G	—	Analyze as soon as possible
Moisture Content	All matrices	—	ASTM D 2216	P, G	—	Analyze as soon as possible

1. Unless otherwise stated, methods are from SW-846.
2. P = Polyethylene; G = Glass; PTFE = Polytetrafluoroethylene.
3. Alternate analytical method for Antimony (7040), Selenium (7740) and Thallium (7841) may be used to achieve detection limits.
4. A separate method for arsenic will only be required if the laboratory cannot achieve the required detection limit using method 6010.
5. The separate methods listed above for arsenic and any other metal with low target detection limits will be used in conjunction with the leaching extraction procedure if the target detection limits for these elements cannot be achieved using method 6010.
6. The analytical laboratory should report the **final pH** of the TCLP/SPLP extract along with the analytical results
7. If refrigeration results in an irreversible physical change to the waste, samples should not be refrigerated.
8. Holding time from sample collection to TCLP analysis. Holding time from TCLP extraction to analysis is as defined elsewhere in the table for the analytical method.
9. There are two extraction fluids identified in the method. For the analysis of waste sampled during this project, Extraction fluid #1 will be used.
10. Holding time is for sludge/solid waste. Holding time for wastewater is 24 hours.
11. Holding time from sample collection to SPLP extraction. Holding time from SPLP extraction to analysis is as defined elsewhere in the table for the analytical method.
12. If Method 7841 is used for thallium analysis, Method 3020 may be used for the preparation/digestion of samples. Method 7740 may be used for both digestion and analysis of selenium. Method 7760 may be used for digestion of silver.

Appendix G
Copies of Chain-of-Custody Forms